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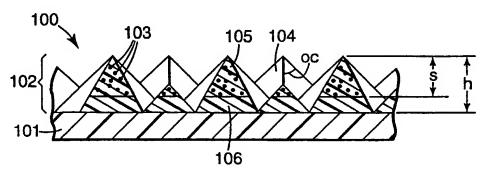
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(54) Title: ABRASIVE MATERIAL HAVING ABRASIVE LAYER OF THREE-DIMENSIONAL STRUCTURE



(57) Abstract: To provide an abrasive material which is excellent in loading resistance and durability, allows no attachments to attach to an abraded surface even when the end surface of the optical fiber is abraded, and is particularly suited for use in abrading a hard material such as an end surface of an optical fiber connector effectively and smoothly into a predetermined shape. The present invention provides an abrasive material for abrading an end surface of an optical fiber connector into a predetermined shape, the abrasive material having a base material (101) and an abrasive layer (102) disposed on the base material, the abrasive layer having a top layer (105) comprising an abrasive composite containing abrasive grains and a binder and a foot portion (106) comprising a binder in the absence of abrasive particles, the abrasive layer having a three-dimensional structure constructed with a plurality of regularly arranged three-dimensional elements (104) having a predetermined shape. Further, the present invention provides a method for producing an abrasive material having an abrasive layer of a three-dimensional structure, the method comprising the steps of: (1) filling a mold sheet having a plurality of regularly arranged recesses, with an abrasive material coating solution containing abrasive grains, a binder, a solvent, to a predetermined depth; (2) removing the solvent from the abrasive material coating solution in the recesses by evaporation; (3) filling the recesses further with a binder; (4) laminating a base material on the mold sheet to bond the binder to the base material; and (5) hardening the binder.



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# ABRASIVE MATERIAL HAVING ABRASIVE LAYER OF THREE-DIMENSIONAL STRUCTURE

#### FIELD OF THE INVENTION

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The present invention relates to an abrasive material having an abrasive layer of a three-dimensional structure, and more particularly to an abrasive material having an abrasive layer of a three-dimensional structure and being suited for abrading an end surface of an optical fiber on which a ferrule is mounted, i.e., an end surface of an optical fiber connector, into a predetermined shape.

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#### **BACKGROUND OF THE INVENTION**

Conventionally, an optical fiber connector which can be easily removed is widely used for connection of optical fibers in an optical fiber communication network. In the connection at the optical fiber connector, end surfaces of the optical fiber ferrules made of an optical fiber and a covering portion (ferrule) for covering the optical fiber are allowed to directly abut each other. Therefore, the optical characteristics at the time of connection, particularly the connection loss, depend on the processing properties and precision of the end surfaces of the optical fibers.

The end surface of the optical fiber ferrule is processed through a number of abrasion steps. The quality of the end surface is influenced by the processing properties and the precision in the final finishing abrasion step. In other words, the major factors for the connection loss of the optical fiber are the degree of finishing roughness of the end surface and its inclination.

With respect to the finishing roughness of an end surface of an optical fiber ferrule, correlation with the particle size of an abrasive material used for the abrasion is reported. For example, in the case of a step index type fiber, the connection loss is about 0.5 dB if the particle size of the abrasive grains is about 1  $\mu$ m, whereas the connection loss is more than about 1.0 dB if the particle size of the abrasive grains is about 15  $\mu$ m.

Upon observation of this correlation, it will be under stood that abrasive grains having a particle size of 10 to 15  $\mu$ m must be used in order to satisfy a standard requiring the connection loss of the optical fiber to be less than 1 dB, and fine grade abrasive grains

having a particle size of less than 1  $\mu m$  must be used in order to satisfy a standard requiring the connection loss of the optical fiber to be less than 0.5 dB.

Japanese Laid-open Patent Publication No. 09-248771/1997 discloses an abrasive tape for an end surface of an optical fiber connector in which the abrasive tape has a base material and an abrasive layer disposed on the base material, the abrasive layer is composed of silica particles having an average particle size of 5 to 30  $\mu$ m and has a binder for connecting these abrasive material particles, and the central line average roughness Ra of the abrasive layer surface is 0.005 to 0.2  $\mu$ m.

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Fine grade abrasive materials such as an abrasive tape for an end surface of an optical fiber connector have a problem of loading. The term "loading" means that the space among abrasive grains is filled with abrasion dusts that protrude to inhibit the abrasive property. For example, in the case where an end surface of an optical fiber connector is abraded, the particles of abrasion dusts stay in the space among the abrasive grains, whereby the cutting ability of the abrasive grains decreases. Further, liquid that is used as a coolant and a lubricant does not act sufficiently between the abrasive material and the end surface of the optical fiber connector, whereby a part of the abrasive layer adheres to the surface of the optical fiber connector after abrasion and its removal is cumbersome.

Moreover, if fine particles are used as the abrasive grains, the period of time required for abrasion will be long. On the other hand, if the particle size of the abrasive grains is increased, the finished end surface of the optical fiber connector will be rough, thereby failing to meet the standard for connection loss of the optical fiber. If both methods are used in combination, the number of abrading steps will increase.

WO92/13680 and WO96/27189 disclose an abrasive material having an abrasive layer of a three-dimensional structure. This abrasive material has a base material and an abrasive layer disposed on the base material, the abrasive layer is made of an abrasive composite containing abrasive particles and a binder, and the abrasive layer has a three-dimensional structure constructed with a plurality of regularly arranged three-dimensional elements having a predetermined shape. As the shape of the three-dimensional elements, tetrahedral shape, pyramidal shape, and others are disclosed.

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This abrasive material is resistant to loading and excellent in durability. However, since the abrasive grains are uniformly dispersed all over in the abrasive layer and the abrasive grains located on the lower part of the abrasive layer do not act effectively, the production cost is high.

Further, an abrasive material having such an abrasive layer of a three-dimensional structure is produced by applying an abrasive slurry containing abrasive particles and a binder in a mold sheet having a structure, superposing a base material on the mold sheet to bond the binder to the base material, hardening the binder by ultraviolet radiation, and removing the mold sheet. In this case, the abrasive slurry must have a sufficient fluidity to be introduced into the structure within the mold sheet. Further, since the ultraviolet radiation is performed after covering the abrasive slurry with the base material, the abrasive slurry must not contain a volatile component.

Therefore, the content of the abrasive grains in the abrasive slurry cannot exceed the critical pigment volume concentration. Accordingly, the conventional abrasive material having an abrasive layer of a three-dimensional structure has a problem that the content of abrasive grains in the abrasive layer cannot be sufficiently raised.

By comparison under abrasive conditions in which the particle size of abrasive grains, the abrasive means, and others are the same, the abrasive property of the abrasive material will decrease as the content of the abrasive grains is reduced. Particularly, in fine grade abrasive materials, the abrasive efficiency will be poor to increase the period of time required for abrasion if the content of the abrasive grains is insufficient.

Accordingly, since the content of the abrasive grains is insufficient, the conventional abrasive material having an abrasive layer of a three-dimensional structure is poor in abrasive property and hence is not suited for abrading a hard material such as an end surface of an optical fiber connector efficiently and smoothly into a predetermined shape.

The present invention has been made to solve the aforesaid problems of the prior art and an object thereof is to provide an abrasive material which is excellent in loading resistance and durability, allows no attachments to attach to an abraded surface even when the end surface of the optical fiber is abraded, and is particularly suited for use in abrading

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a hard material such as an end surface of an optical fiber connector effectively and smoothly into a predetermined shape.

#### SUMMARY OF THE INVENTION

The present invention provides an abrasive material for abrading an end surface of an optical fiber connector into a predetermined shape, the abrasive material having a base material and an abrasive layer disposed on the base material, the abrasive layer having an abrasive composite containing abrasive grains and a binder as construction components, the abrasive layer having a three-dimensional structure constructed with a plurality of regularly arranged three-dimensional elements having a predetermined shape, thereby to achieve the aforesaid object of the present invention.

Further, the present invention provides a method for producing an abrasive material having an abrasive layer of a three-dimensional structure, the method comprising the steps of: (1) filling a mold sheet having a plurality of regularly arranged recesses, with an abrasive material coating solution containing abrasive grains, a binder, and a solvent, to a predetermined depth; (2) removing the solvent from the abrasive material coating solution in the recesses by evaporation; (3) filling the recesses further with a binder; (4) laminating a base material on the mold sheet to bond the binder to the base material; and (5) hardening the binder.

The abrasive material having an abrasive layer of a three-dimensional structure is preferably produced by the aforesaid production method.

#### BRIEF DESCRIPTION OF THE DRAWINGS

The above and further objects and features of the invention will more fully be apparent from the following detailed description with accompanying drawings, in which,

Fig. 1 is a section view illustrating an abrasive material having an abrasive layer of a three-dimensional structure according to an embodiment of the present invention;

Fig. 2 is a top view illustrating an abrasive material having an abrasive layer of a three-dimensional structure according to an embodiment of the present invention;

Fig. 3 is a top view illustrating an abrasive material having an abrasive layer of a three-dimensional structure according to an embodiment of the present invention;

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Fig. 4 is a perspective section view illustrating an abrasive material having an abrasive layer of a three-dimensional structure according to an embodiment of the present invention;

- Fig. 5 is a top view illustrating an abrasive material having an abrasive layer of a three-dimensional structure according to an embodiment of the present invention;
- Figs. 6(a) to 6(e) are model views illustrating steps for producing an abrasive material having an abrasive layer of a three-dimensional structure;
- Fig. 7 is a graph showing change with time of an abraded amount when an end surface of an optical fiber connector is abraded with various abrasive materials;
- Fig. 8 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;
- Fig. 9 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;
- Fig. 10 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the prior art;
- Fig. 11 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the prior art;
- Fig. 12 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the prior art;
- Fig. 13 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;
- Fig. 14 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;
- Fig. 15 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the prior art;
- Fig. 16 is a graph showing change with time of an abraded amount when a circular rod of zirconia is abraded with various abrasive materials;
- Fig. 17 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;
- Fig. 18 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;

Fig. 19 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;

Fig. 20 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;

Fig. 21 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the prior art;

Fig. 22 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the prior art;

Fig. 23 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention;

Fig. 24 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention; and

Fig. 25 is a microscope photograph of an end surface of an optical fiber connector after being abraded with the abrasive material of the present invention.

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## DETAILED DESCRIPTION OF THE INVENTION

Fig. 1 is a section view illustrating an abrasive material having an abrasive layer of a three-dimensional structure as an embodiment of the present invention. An abrasive material 100 has a base material 101 and an abrasive layer 102 disposed on a surface of the base material 101.

Preferable examples of the base material for the present invention include polymer film, paper, cloth, metal film, vulcanized fiber, non-woven base material, a combination thereof, and a processed product thereof. In the case of spherically abrading the end surface of the optical fiber connector, the base material is preferably flexible, thereby facilitating formation of a spherical shape. The base material is preferably transparent with respect to ultraviolet radiation, since it is convenient in the production process.

The base material may be, for example, a polymer film such as polyester film. The polymer film may be undercoated with a material such as polyethylene acrylic acid in order to promote bonding to the base material of the abrasive composite.

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The abrasive layer 102 has an abrasive composite containing a matrix of a binder and abrasive grains 103 dispersed therein as construction components.

The abrasive composite is formed from a slurry containing a plurality of abrasive grains dispersed in the binder which is in an unhardened or ungelated state. In hardening or gelation, the abrasive composite is solidified, i.e. is fixed to have a predetermined shape and a predetermined structure.

The dimension of the abrasive grains may vary depending on the type of the abrasive grains or the intended use of the abrasive material. For example, the dimension is 0.01 to 1 µm, preferably 0.01 to 0.5 µm, more preferably 0.01 to 0.1 µm for the final finishing abrasion, and is 0.5 to 20 µm, preferably 0.5 to 10 µm for rough abrasion in forming a curved surface. Preferable examples of the abrasive grains for the present invention include diamond, cubic boron nitride, cerium oxide, fused aluminum oxide, heat-treated aluminum oxide, sol-gel aluminum oxide, silicon carbide, chromium oxide, silica, zirconia, alumina zirconia, iron oxide, garnet, and a mixture thereof. Especially preferable ones are diamond, cubic boron nitride, aluminum oxide, and silicon carbide for the rough abrasion, and silica and aluminum oxide for the finishing abrasion.

The binder is hardened or gelated to form an abrasive layer. Preferable examples of the binder include phenolic resin, resol-phenolic resin, aminoplast resin, urethane resin, epoxy resin, acrylate resin, polyester resin, vinyl resin, melamine resin, acrylated isocyanurate resin, urea-formaldehyde resin, isocyanurate resin, acrylated urethane resin, acrylated epoxy resin, and a mixture thereof. The binder may be a thermoplastic resin. Especially preferable examples of the binder include phenolic resin resol-phenolic resin, epoxy resin, and urethane resin.

The binder may be radiation-curing. The radiation-curing binder is a binder that is at least partially hardened or is at least partially polymerizable by radiation energy. Depending on the binder to be used, an energy source such as heat, infrared radiation, electron beam radiation, ultraviolet radiation, or a visible light radiation is used.

Typically, these binders are polymerized by a free radical mechanism. Preferably, these binders are selected from the group consisting of acrylated urethane, acrylated epoxy, an aminoplast derivative having an  $\alpha$ ,  $\beta$  unsaturated carbonyl group, an ethylenic

unsaturated compound, an isocyanurate derivative having at least one acrylate group, isocyanate having at least one acrylate group, and a mixture thereof.

If the binder is hardened by ultraviolet radiation, a photoinitiator is required to start free radical polymerization. Preferable examples of the photoinitiator to be used for this purpose include organic peroxides, azo compounds, quinones, benzophenones, nitroso compounds, acryl halides, hydrazones, mercapto compounds, pyrylium compounds, triacrylimidazole, bisimidazole, chloroalkyltriazine, benzoin ether, benzyl ketal, thioxanthone, and acetophenone derivatives. A preferable photoinitiator is 2,2-dimethoxy-1,2-diphenyl-1-ethanone.

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If the binder is hardened by visible light radiation, it is necessary that a photoinitiator stakets a free radical polymerization. Preferable examples of the photoinitiator for this purpose are disclosed in United States Patent No. 4,735,632, column 3, line 25 to column 4, line 10, column 5, lines 1 to 7, and column 6 lines 1 to 35, which are incorporated herein by reference.

The weight proportion of the abrasive grains to the binder is typically within a range of about 1.5 to 10 parts of abrasive grains with respect to one part of the binder, preferably about 2 to 7 parts by weight of abrasive grains with respect to one part of the binder. This proportion may vary depending on the size of the abrasive grains, the type of the binder to be used, and the intended purpose of the abrasive material.

In smoothly and finely abrading a hard material such as an end surface of an optical fiber connector, the concentration of the abrasive grains contained in the abrasive composite is preferably within a range of 43 to 90 wt% if the abrasive grains are made of silicon carbide; 70 to 90 wt% if the abrasive grains are made of spherical abrasive particles of alumina, silica, or the like; 37 to 90 wt% if the abrasive grains are made of alumina; and 39 to 90 wt% if the abrasive grains are made of diamond.

The abrasive composite may contain a material other than the abrasive grains and the binder. For example, the abrasive material may contain ordinary additives such as a coupling agent, a lubricant, a dye, a pigment, a plasticizer, a filler, a stripping agent, an abrasive aid, and a mixture thereof.

The abrasive composite can contain a coupling agent. Addition of the coupling agent can considerably reduce the covering viscosity of a slurry to be used for formation of

the abrasive composite. Preferable examples of the coupling agent for the present invention include organic silane, zircoaluminate, and titanate. The amount of the coupling agent is typically less than 5 wt%, preferably less than 1 wt%, of the binder.

The abrasive layer 102 has a three-dimensional structure constructed with a plurality of regularly arranged three-dimensional elements 104 having a predetermined shape. The three-dimensional elements 104 each have a tetrahedral shape in which ridges are connected at a point on the top. In this case, the angle  $\alpha$  formed between two ridges is typically 30 to 150°, preferably 45 to 140°. The three-dimensional elements 104 may have a pyramidal shape. In this case, the angle  $\alpha$  formed between two ridges is typically 30 to 150°, preferably 45 to 140°.

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The points on the top of the three-dimensional elements 104 are located on a plane parallel to the surface of the base material substantially over an entire region of the abrasive material. In Fig. 1, the symbol h represents the height of the three-dimensional elements 104 from the surface of the base material. The height h is typically 2 to 300  $\mu$ m, preferably 5 to 150  $\mu$ m. The variation of the height of the points on the top is preferably less than 20%, more preferably less than 10%, of the height of the three-dimensional elements.

The three-dimensional elements 104 are arranged in a predetermined configuration. In Fig. 1, the three-dimensional elements 104 are most closely packed. Typically the three-dimensional elements are repeated with a predetermined period. This repetitive shape is one-directional or preferably two-directional.

The abrasive grains do not protrude beyond the surface of the shape of the three-dimensional elements. In other words, the three-dimensional elements 104 are constructed with flat planes. For example, the surfaces constituting the three-dimensional elements 104 have a surface roughness Ra of less than 2  $\mu$ m, preferably less than 1  $\mu$ m.

In the three-dimensional element 104, its top portion 105 performs an abrading function. While the abrasive material is subjected to abrasion, the three-dimensional elements are decomposed starting from the top portion, thereby allowing unused abrasive grains to appear. Therefore, in order to increase the abrasive property of the abrasive material, the concentration of the abrasive grains in the abrasive composite located in the top portion of the three-dimensional element is preferably increased to be as high as

possible so that the abrasive material may have a higher abrasive property to be suited for abrading a hard material. The concentration of the abrasive grains in the abrasive composite located in the top portion of the three-dimensional element more preferably exceeds the critical pigment volume concentration.

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Generally, the critical pigment volume concentration is considered to be the pigment volume concentration where there is just sufficient binder to coat pigment surfaces and provide a continuous phase throughout the film. The critical pigment volume concentration as used herein means a volume concentration of abrasive grains when the gaps among the grains are just filled with a binder. In the case where the binder is liquid, the mixture has fluidity if the concentration is less than the critical pigment volume concentration, whereas the mixture loses its fluidity if the concentration exceeds the critical pigment volume concentration. If the concentration of the abrasive grains in the abrasive composite located in the top portion of the three-dimensional element is less than or equal to the critical pigment volume concentration, the abrasive property of the abrasive material will be insufficient, so that the abrasive material will not be suitable for abrasion of a hard material such as an end surface of an optical fiber connector.

The foot portion 106 of the three-dimensional element, namely the lower portion of the abrasive layer adhering to the base material, does not usually perform an abrading function. This is because, if the abrasive layer is worn to the lower portion, the abrasive material is usually discarded. The foot portion 106 of the three-dimensional element that does not perform the abrading function need not contain abrasive grains, so that the foot portion 106 may be made of the binder alone.

By allowing the three-dimensional element 104 to have such a two-layer structure, the amount of the comparatively expensive abrasive grains can be saved, whereby the abrasive material can be provided at a lower cost. In addition, since the binder in the foot portion 106 can be designed considering only the adhesive power of the binder to the base material, poor adhesion to the base material hardly occurs.

In Fig. 1, the symbol s represents the height of the top portion 105 of the three-dimensional element. The height s is, for example, 5 to 95%, preferably 10 to 90%, of the height h of the three-dimensional element.

Fig. 2 is a top view of this abrasive material. In Fig. 2, the symbol o represents the bottom side length of the three-dimensional element. The symbol p represents the distance between the tops of adjacent three-dimensional elements. The length o is, for example, 5 to  $1000 \, \mu m$ , preferably 10 to  $500 \, \mu m$ . The distance p is, for example, 5 to  $1000 \, \mu m$ , preferably  $10 \, to \, 500 \, \mu m$ .

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In another embodiment, the three-dimensional element may have a tetrahedral or pyramidal shape whose top is truncated to a predetermined height. In this case, the top of the three-dimensional element is preferably formed of a triangular or quadrangular plane parallel to the surface of the base material, and substantially all of these planes are preferably located on a plane parallel to the surface of the base material. The height of the three-dimensional element is 5 to 95%, preferably 10 to 90%, of the height h of the three-dimensional element before truncation of the top. Fig. 3 is a top view of the abrasive material according to this embodiment.

In Fig. 3, the symbol o represents the bottom side length of the three-dimensional element. The symbol u represents a distance between bottom sides of adjacent three-dimensional elements. The symbol y represents the length of one side of the top plane. The length o is, for example, 5 to 2000  $\mu$ m, preferably 10 to 1000  $\mu$ m. The distance u is, for example, 0 to 1000  $\mu$ m, preferably 2 to 500  $\mu$ m. The length y is, for example, 0.5 to 1800  $\mu$ m, preferably 1 to 900  $\mu$ m.

Fig. 4 is a perspective section view of an abrasive material having an abrasive layer of a three-dimensional structure according to another embodiment of the present invention. An abrasive material 400 is an abrasive material having a base material 401 and an abrasive layer 402 disposed on the surface of the base material.

The abrasive layer 402 has an abrasive composite containing a matrix of a binder and abrasive grains 403 dispersed therein as construction components.

The abrasive layer 402 has a three-dimensional structure constructed with a plurality of regularly arranged three-dimensional elements having a predetermined shape. The three-dimensional elements 404 has a prismatic shape formed of a laterally-placed triangular prism. The angle  $\beta$  of the three-dimensional element 404 is typically 30 to 150°, preferably 45 to 140°.

The ridges on the top of the three-dimensional elements 404 are located on a plane parallel to the surface of the base material substantially over an entire region of the abrasive material. In Fig. 4, the symbol h represents the height of the three-dimensional element from the surface of the base material. The height h is typically 2 to 600  $\mu$ m, preferably 4 to 300  $\mu$ m. The variation of the height of the top lines is preferably less than 20%, more preferably less than 10%, of the height of the three-dimensional element 404.

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Like the three-dimensional element 104, the three-dimensional element 404 preferably has a two-layer structure including a top portion 405 made of an abrasive composite and a foot portion 406 made of a binder. In Fig. 4, the symbol's represents the height of the top portion of the three-dimensional element. The height's is, for example, 5 to 95%, preferably 10 to 90%, of the height h of the three-dimensional element.

Typically, the three-dimensional elements 404 are arranged in a stripe pattern. In Fig. 4, the symbol w represents the length of the short bottom side of the three-dimensional element (width of the three-dimensional element). The symbol p represents the distance between tops of adjacent three-dimensional elements. The symbol u represents the distance between long bottom sides of adjacent three-dimensional elements. The length w is, for example, 2 to 2000  $\mu$ m, preferably 4 to 1000  $\mu$ m. The distance p is, for example, 2 to 4000  $\mu$ m, preferably 4 to 2000  $\mu$ m. The distance u is, for example, 0 to 2000  $\mu$ m, preferably 0 to 1000  $\mu$ m.

The length of the three-dimensional element may extend substantially over an entire region of the abrasive material. Alternatively, the length of the three-dimensional element may be cut to a suitable length. The ends of the three-dimensional elements may be either aligned or non-aligned. The ends of the prismatic three-dimensional elements may be cut at an acute angle from its bottom to form a house shape having four inclined surfaces. Fig. 5 is a top view of the abrasive material according to this embodiment.

In Fig. 5, the symbol 1 represents the length of a long bottom side of the three-dimensional element. The symbol v represents the distance of a portion of the three-dimensional element cut at an acute angle. The symbol x represents the distance between short bottom sides of adjacent three-dimensional elements. The symbols w, p, and u have the same meaning as in Fig. 4. The length 1 is, for example, 5 to 10000  $\mu m$ , preferably 10 to 5000  $\mu m$ . The distance v is, for example, 0 to 2000  $\mu m$ , preferably 1 to

1000  $\mu m$ . The distance x is, for example, 0 to 2000  $\mu m$ , preferably 0 to 1000  $\mu m$ . The length w is, for example, 2 to 2000  $\mu m$ , preferably 4 to 1000  $\mu m$ . The distance p is, for example, 2 to 4000  $\mu m$ , preferably 4 to 2000  $\mu m$ . The distance u is, for example, 0 to 2000  $\mu m$ , preferably 0 to 1000  $\mu m$ .

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The abrasive material having an abrasive layer of a three-dimensional structure of the present invention exemplified in Figs. 1 to 5 are particularly suited for use in abrading an end surface of an optical fiber connector, and can provide an end surface of an optical fiber connector with an extremely small connection loss. For example, the abrasive material having an abrasive layer of a three-dimensional structure according to the present invention provides an end surface of an optical fiber connector with a connection loss of less than 1.0 dB, or less than 0.5 dB.

The abrasive material of the present invention is preferably produced by the method described below.

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First, an abrasive slurry is prepared which contains abrasive grains, a binder, and a solvent. The abrasive slurry to be used herein is a composition containing the binder, the abrasive grains, and optional additives such as a photoinitiator in sufficient amounts to constitute an abrasive composite and further containing a volatile solvent in a sufficient amount to impart fluidity to the mixture. Even if the content of the abrasive grains in the abrasive composite exceeds the critical pigment volume concentration, the fluidity can be maintained by allowing the abrasive slurry to contain a volatile solvent.

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A preferable volatile solvent is an organic solvent that dissolves the binder and shows volatility at room temperature to 170°C. Specific examples of the organic solvent include methyl ethyl ketone, methyl isobutyl ketone, toluene, xylene, ethanol, isopropyl alcohol, ethyl acetate, butyl acetate, tetrahydrofuran, propylene glycol monomethyl ether, and propylene glycol monomethyl ether acetate. Another preferable solvent is water.

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Next, a mold sheet is prepared which has a plurality of regularly arranged recesses tapered toward the bottom. The shape of the recesses may be an inverse of the three-dimensional elements to be formed. The mold sheet may be made of a metal such as nickel or plastics such as polypropylene. For example, a thermoplastic resin such as polypropylene is preferable because it can be embossed at its melting point on a metal tool to form recesses of a predetermined shape. Further, if the binder is a radiation-curing type

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resin, it is preferable to use a material that transmits ultraviolet rays and visible light. Figs. 6(a) to 6(e) are model views showing steps for producing an abrasive material having an abrasive layer of a three-dimensional structure.

Referring to Fig. 6(a), the obtained mold sheet 601 is filled with an abrasive slurry 602. The amount of the abrasive slurry to be used in filling the mold sheet is such that it can form a top portion 105, 405 after the solvent is evaporated and the binder is hardened. Typically, the amount of the abrasive slurry may be such that its depth from the bottom is a dimension s shown in Figs. 1 and 4 after the evaporation of the solvent.

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The mold sheet can be filled with the abrasive slurry by applying the abrasive slurry onto the mold sheet by means of a coating apparatus such as a roll coater. The viscosity of the abrasive slurry for application is preferably adjusted to be 10 to 106 cps, particularly 100 to 105 cps.

Referring to Fig. 6(b), the solvent is evaporated and removed from the abrasive slurry. In doing this, the mold sheet filled with the abrasive slurry is heated to 50 to 150°C for 0.2 to 10 minutes. If the binder is a thermoplastic resin, the mold sheet may be heated at its curing temperature for simultaneously performing a hardening step. If the volatility of the solvent is high, the mold sheet may be left to stand at room temperature for several minutes to several hours.

Referring to Fig. 6(c), the mold sheet is further filled with a binder 603 for lamination to fill the recesses with the binder. The lamination binder may be the same as or different from the one used in preparing the abrasive slurry. A binder having a good adhesion to the base material is preferable as the lamination binder.

Preferable examples of the lamination binder is acrylate resin, epoxy resin, and urethane resin. The mold sheet may be filled with the lamination binder in the same manner as the abrasive slurry.

Referring to Fig. 6(d), a base material 604 is superposed on the mold sheet 601 to allow the binder to adhere to the base material. The adhesion is carried by pressing with a roll for lamination.

The binder is hardened. The term "hardening" as used herein means that the binder is polymerized into a solid state. After the hardening, the specific shape of the abrasive layer does not change. The hardening of the binder in the abrasive slurry and the

hardening of the lamination binder introduced alone at the later step may be performed either separately or simultaneously.

The binder is hardened by heat, infrared radiation, or by electron beam radiation, ultraviolet radiation, or by another radiation energy such as visible light radiation. The amount of radiation energy to be applied may vary depending on the type of the binder and the radiation energy source. Usually, those skilled in the art can suitably determine the amount of radiation energy to be applied. The period of time required in hardening may vary depending on the thickness, density, temperature of the binder, the properties of the composition, and others.

For example, the binder may be hardened by radiating ultraviolet rays (UV) from above the transparent base material.

Referring to Fig. 6(e), the mold sheet is removed to produce an abrasive material 606 composed of the base material 604 and the abrasive layer 605 having a three-dimensional structure. The binder may be hardened after the mold sheet is removed.

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#### Examples

The present invention will be described in more detail by way of the following examples. However, the present invention is not limited by these examples.

#### 20 Example 1

An abrasive material coating solution was prepared by mixing the components shown in Table 1.

Table 1

		Non-volatile	Weight after
Components	Weight (g)	component (%)	drying (g)
Diamond abrasive grains	100.000	100.000	100.000
(particle size : 2 to 4 μm) "LS600F 2-4"			
(Lands Superabrasives, Co.)			
Epoxy resin "YD-20N" (Toto Kasei Co.,	17.500	50.000	8.750
Ltd.)			
(as 50% MEK solution)			
Urethane resin solution "EA-1443"	29.545	55.000	16.250
(Daicel Kagaku Kogyo Co., Ltd.)			
Methyl ethyl ketone (MEK)	75.000	0.000	0.000

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Aerosol AY	1.000	50.000	0.500
(AMERICAN CYANAMID			
COMPANY)			
(as 50% MEK solution)			0.400
Polyfunctional isocyanate "Coronate L"	12.564	75.000	9.423
(Nippon Polyurethane Kogyo Co., Ltd.)			
Total	235.609	57.266	134.923

Abrasive grains/binder proportion = 2.91 Abrasive grains/(binder + additives) proportion = 2.86

A lamination binder was prepared by mixing the components shown in Table 2.

Table 2

	Weight	Non-volatile	Weight
Components	(g)	component (%)	after drying
1			(g)
Monoacrylate monomer "KAYARAD R-564"	50.000	100.000	50.000
(Nippon Kayaku Co., Ltd.)			
Diacrylate monomer "KAYARAD R-551"	50.000	100.000	50.000
(Nippon Kayaku Co., Ltd.)			
Benzophenone	4.000	100.000	4.000
1,4-diazabicyclo[2.2.2]octane	1.000	100.000	1.000
(DABCO)			
Total	105.000	400	105.000

A mold sheet made of polypropylene and having recesses with a shape of inverted three-dimensional elements shown in Fig. 4 was prepared. An abrasive slurry was applied onto the mold sheet by means of a roll coater and dried at  $50^{\circ}$ C for 5 minutes. A lamination binder was applied thereon and further a transparent polyester film having a thickness of 75  $\mu$ m was superposed and pressed by a roll for lamination. Ultraviolet rays were radiated from the polyester film side to harden the lamination binder. Subsequently, the binder of the abrasive slurry was hardened by heating at  $70^{\circ}$ C for 24 hours.

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The mold sheet was removed and the resultant was cooled to room temperature to produce an abrasive material. In the abrasive material, the abrasive layer has a three-dimensional structure having a prismatic shape arranged in a stripe pattern shown in Fig. 4. The dimensions thereof are shown in Table 3.

Table 3

Symbol	Dimension (µm)
h	25
S	15
w	50
р	50
u	0
β	90°

This abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk. An end surface of an optical connector ferrule was abraded with the use of the obtained abrasive disk. The abrasion conditions are shown in Table 4.

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Table 4

Abrader	"OFL-12" made by Seiko Denshi Kogyo Co., Ltd.
Load	Point 2 (about 1.5 kg/cm <sup>2</sup> )
Cooling liquid	Purified water
Number of abraded samples	12

The change with time of the abraded amount is shown in Fig. 7. After the abrasion, the end surface of the optical connector ferrule was observed by an electron microscope, whereby a smooth surface was confirmed. The obtained microscope photograph is shown in Fig. 8.

Example 2

An abrasive slurry was prepared by mixing the components shown in Table 5.

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Table 5

	Weight	Non-volatile	Weight after
Components	(g)	component (%)	drying (g)
Diamond abrasive grains	150.000	100.000	150.000
(particle size : 2 to 4 μm) "LS600F 2-4"			
(Lands Superabrasives, Co.)			
Epoxy resin solution "YDCN-703PEK75"	66.670	75.000	50.003
(Toto Kasei Co., Ltd.)			
Methyl ethyl ketone (MEK)	40.500	0.000	0.000
Propylene glycol monomethyl ether	34.500	0.000	0.000
(PGM)	,		
2-methylimidazole (2MZ)	12.501	20.000	2.500
(20% propylene glycol monomethyl ether			
solution)			
Total	304.171	66.575	202.503

Abrasive grains/binder proportion = 2.86

Abrasive grains/(binder + additives) proportion = 2.86

An abrasive disk was fabricated in the same manner as in Example 1 except that this abrasion slurry was used, and an end surface of an optical connector ferrule was abraded. The change with time of the abraded amount is shown in Fig. 7. After the abrasion, the end surface of the optical connector ferrule was observed by an electron microscope, whereby a smooth surface was confirmed. The obtained microscope photograph is shown in Fig. 9.

# Comparative Example 1

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An abrasive material "Imperial Sign Diamond Lapping Film 3 Mil 3 Micron Type H" made by Minnesota Mining and Manufacturing Co., Ltd. was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk. An end surface of an optical connector ferrule was abraded in the same manner as in Example 1 except that this abrasive disk was used. The change with time of the abraded amount is shown in Fig. 7. After the abrasion, the end surface of the optical connector ferrule was observed by an electron microscope, whereby a rough surface was confirmed. The obtained microscope photograph is shown in Fig. 10.

## Comparative Example 2

The abrasive slurry prepared in Example 1 was applied onto a polyester film having a thickness of 75 µm by means of a knife coater and the solvent was removed by evaporation to form an abrasive layer having a thickness of 11 µm. The abrasive layer was heated at 70°C for 24 hours to harden the binder. The obtained abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

An end surface of an optical connector ferrule was abraded in the same manner as in Example 1 except that this abrasive disk was used. The change with time of the abraded amount is shown in Fig. 7. After the abrasion, the end surface of the optical connector ferrule was observed by an electron microscope, whereby a rough surface was confirmed. The obtained microscope photograph is shown in Fig. 11.

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# Comparative Example 3

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The abrasive slurry prepared in Example 2 was applied onto a polyester film having a thickness of 75 µm by means of a knife coater and the solvent was removed by evaporation to form an abrasive layer having a thickness of 11 µm. The abrasive layer was heated at 70°C for 24 hours to harden the binder. The obtained abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

An end surface of an optical connector ferrule was abraded in the same manner as in Example 1 except that this abrasive disk was used. The change with time of the abraded amount is shown in Fig. 7. After the abrasion, the end surface of the optical connector ferrule was observed by an electron microscope, whereby a rough surface was confirmed. The obtained microscope photograph is shown in Fig. 12.

By comparing Figs. 8 and 9 with Fig. 10, it will be understood that the abrasive materials of Examples 1 and 2 give a more smooth abraded surface than the abrasive material of Comparative Example 1 which is a current product. Also, by comparing Fig. 8 with Fig. 11, it will be understood that the abrasive material of Example 1 gives a more smooth surface than the abrasive material of Comparative Example 2 which is an abrasive material made of the same slurry but having an abrasive layer without a three-dimensional structure. By comparing Fig. 9 with Fig. 12, it will be understood that the abrasive material of Example 2 gives a more smooth surface than the abrasive material of Comparative Example 3 which is an abrasive material made of the same slurry but having an abrasive layer without a three-dimensional structure.

From the graph shown in Fig. 7, it will be understood that the abrasive disk of Example 2 exhibits a higher abrasive property than the abrasive disks of Comparative Examples 1 to 3.

#### Example 3

An abrasive slurry was prepared by mixing the components shown in Table 6.

Table 6

	Weight	Non-volatile	Weight after
Components	(g)	component (%)	drying (g)
Colloidal silica "Snowtechs IPA-ST"	100.00	30.000	30.00
(Nissan Kagaku Kogyo Co., Ltd.)			
Diacrylate monomer "KAYARAD HDDA"	15.00	100.000	15.00
(Nippon Kayaku Co., Ltd.)			
Photoinitiator "Irgacure 369" (CIBA-GEIGY)	0.30	100.000	0.30
Total	115.30	46.030	45.30

Abrasive grains/binder proportion = 2.00 Abrasive grains/(binder + additives) proportion = 1.96

The same mold sheet made of polypropylene as used in Example 1 was prepared. An abrasive slurry was applied onto the mold sheet by means of a roll coater and dried at 60°C for 5 minutes. A lamination binder prepared in Example 1 was applied thereon and further a transparent polyester film having a thickness of 75 µm was superposed and pressed by a roll for lamination. Ultraviolet rays were radiated from the polyester film side to harden the binder. The mold sheet was removed and the resultant was cooled to room temperature to produce an abrasive material. This abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

In the meantime, an optical connector ferrule was prepared and an end surface thereof was abraded under the same abrasion condition as in Table 7 with the use of an abrasive material "Imperial Sign Diamond Lapping Film 3 Mil 0.5 Micron Type H" made by Minnesota Mining and Manufacturing Co., Ltd. An end surface of this optical connector ferrule was further abraded with the use of the fabricated abrasive disk. The abrasion condition is shown in Table 7.

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Table 7

Abrader	"OFL-12" made by Seiko Denshi Kogyo Co., Ltd.
Load	Point 3 (about 2 kg/cm <sup>2</sup> )
Number of abraded samples	6

After the abrasion, the end surface of the optical connector ferrule was observed by an electron microscope, whereby a smooth surface was confirmed. The obtained microscope photograph is shown in Fig. 13.

The shape of the end surface of the optical connector ferrule after the abrasion was measured by means of "Zoom Interferometer ZX-1 Mini PMS" made by Direct Optical Research Company (DORC), and the reflected damping amount was measured by means of "Back Reflection Meter RM300A" made by JDS FITEL. The results are shown in Table 9.

#### Example 4

An abrasive slurry was prepared by mixing the components shown in Table 8.

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Table 8

	Weight	Non-volatile	Weight after
Components	(g)	component (%)	drying (g)
Colloidal silica "Snowtechs IPA-ST"	100.00	30.000	30.00
(Nissan Kagaku Kogyo Co., Ltd.)			
Diacrylate monomer "KAYARAD HDDA"	7.50	100.000	7.50
(Nippon Kayaku Co., Ltd.)			
Monoacrylate monomer "KAYARAD R-564"	7.50	100.000	7.50
(Nippon Kayaku Co., Ltd.)			
Photoinitiator "Irgacure 369" (CIBA-GEIGY)	0.30	100.000	0.30
Total	115.30	46.030	45.30

Abrasive grains/binder proportion = 2.00

Abrasive grains/(binder + additives) proportion = 1.96

An abrasive disk was fabricated in the same manner as in Example 3 except that this abrasive slurry was used, and an end surface of an optical connector ferrule was abraded. A microscope photograph of the end surface after the abrasion is shown in Fig. 14. The shape of the end surface and the reflected damping amount are shown in Table 9.

#### 20 Comparative Example 4

An abrasive material "Imperial Sign Diamond Lapping Film 3 Mil 0.05 Micron AO Type P" made by Minnesota Mining and Manufacturing Co., Ltd. was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk. An end surface of an optical connector ferrule was abraded in the same manner as in Example 3 except that this abrasive disk was used. A microscope photograph of the end surface after

the abrasion is shown in Fig. 15. The shape of the end surface and the reflected damping amount are shown in Table 9.

# Comparative Example 5

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The abrasive slurry prepared in Example 3 was applied onto a polyester film having a thickness of 75  $\mu$ m by means of a knife coater and the solvent was removed by evaporation to form an abrasive layer having a thickness of 4  $\mu$ m. A polyester film having a thickness of 31  $\mu$ m was laminated on this abrasive layer, and the binder was hardened by radiating ultraviolet rays. The obtained abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

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An end surface of an optical connector ferrule was abraded in the same manner as in Example 3 except that this abrasive disk was used. However, attachments accumulated on the end surface during the abrasion, making it impossible to perform effective abrasion.

#### Comparative Example 6

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The abrasive slurry prepared in Example 4 was applied onto a polyester film having a thickness of 75  $\mu$ m by means of a knife coater and the solvent was removed by evaporation to form an abrasive layer having a thickness of 4  $\mu$ m. A polyester film having a thickness of 31  $\mu$ m was laminated on this abrasive layer, and the binder was hardened by radiating ultraviolet rays. The obtained abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

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An end surface of an optical connector ferrule was abraded in the same manner as in Example 3 except that this abrasive disk was used. However, attachments accumulated on the end surface during the abrasion, making it impossible to perform effective abrasion.

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Table 9

							Compa	rativ
Abrasive material samples	Exam	ple 3	Examj	ole 4	Examp	ole 4	e Exam	iple 4
Coolant	Pure v	vater	Pure v	vater	2-prop	anol	Pure v	vater
Measurement parameters	Mean	σ	Mean	σ	Mean	σ	Mean	σ
Radius of curvature (mm)	15.10	1.56	17.15	3.90	18.91	5.05	14.73	0.84
Fiber height	28.06	7.7	9.4	8.4	-60.3	34.9	-31.5	3.9
(Spherical Fit: nm)						,		
Fiber height	163.0	20.7	132.9	38.9	52.9	10.1	106.1	8.7
(Planar Fit: nm)								
Diameter (m)	126.9	0.3	126.7	0.3	126.6	0.3	127.3	0.4
Reflected damping amount	46.1	0.2	44.7	1.1	47.3	2.2	41.7	0.5
(dB)				<u> </u>			L	

As shown in Figs. 13 and 14, when the abrasive materials of Examples 3 and 4 were used, the abrasion streaks created by abrasion with the abrasive material "Imperial Sign Diamond Lapping Film 3 Mil 0.5 Micron Type H" made by Minnesota Mining and Manufacturing Co., Ltd. (Fig. 10) disappeared by abrasion of 60 seconds. This end surface of the optical connector ferrule had been extremely smoothly and finely abraded and, as shown in Table 9, the reflected damping amount was extremely small compared with Comparative Example 4. The abrasive material of Example 4 showed an extremely good result when abrasion was performed with the use of 2-propanol as the cooling liquid.

#### Example 5

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An abrasive slurry was prepared by mixing the components shown in Table 10.

Table 10

	Weight	Non-volatile	Weight after
Components	(g)	component (%)	drying (g)
Diamond abrasive grains	100.000	100.000	100.000
(particle size : 7 to 10 μm) "LS600F 7-10"			,
(Lands Superabrasives, Co.)			
Epoxy resin solution "YDCN-703PEK75"	46.667	75.000	35.000
(Toto Kasei Co., Ltd.)			
Methyl ethyl ketone (MEK)	40.000	0.000	0.000
Aerosol AY	1.000	50.000	0.500
(AMERICAN CYANAMID COMPANY)			
(as 50% MEK solution)			
2-methylimidazole (2MZ)	8.750	20.000	1.750
(20% propylene glycol monomethyl ether			
solution)			
Total	196.417	69.877	137.25

Abrasive grains/binder proportion = 2.72 Abrasive grains/(binder + additives) proportion = 2.69

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The same mold sheet made of polypropylene as used in Example 1 was prepared. An abrasive slurry was applied onto the mold sheet by means of a roll coater and dried at 70°C for 5 minutes. A lamination binder prepared in Example 1 was applied thereon and further a transparent polyester film having a thickness of 75 µm was superposed and pressed by a roll for lamination. Ultraviolet rays were radiated from the polyester film side to harden the lamination binder. Subsequently, the binder in the abrasive slurry was hardened by heating at 70°C for 24 hours.

The resultant was cooled to room temperature and the mold sheet was removed to produce an abrasive material. This abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

A circular rod of zirconia (diameter: 3 mm) was abraded with the use of the fabricated abrasive disk. The abrasion conditions are shown in Table 11.

Table 11

Abrader	"OFL-12" made by Seiko Denshi Kogyo Co., Ltd.
Load	Point 1 (about 2.5 kg/cm <sup>2</sup> )
Cooling liquid	Purified water
Number of abraded samples	6

The change with time of the abraded amount is shown in Fig. 16.

Subsequently, the abrasive disk was replaced with a new one, and the end surface of the optical connector ferrule was abraded. The abrasion condition is shown in Table 12.

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Table 12

Abrader	"OFL-12" made by Seiko Denshi Kogyo Co Ltd.
Load	Point 1 (about 2.5 kg/cm <sup>2</sup> )
Cooling liquid	Purified water
Number of abraded samples	12

After the abrasion, the end surface of the optical connector ferrule was observed by an electron microscope, whereby a smooth surface was confirmed. A microscope photograph is shown in Fig. 17.

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# Example 6

An abrasive material was fabricated in the same manner as in Example 5 except that a mold sheet made of polypropylene and having recesses with a shape of inverted three-dimensional elements shown in Fig. 5 was used. In this abrasive material, the abrasive layer has a three-dimensional structure of a house shape arranged in a stripe pattern as shown in Fig. 5. The dimensions are shown in Table 13.

Table 13

Symbol	Dimension (µm)
h*	20 μm
s*	14 μm
w	40 μm
Р	50 μm
u	10 μm
l	280 μm
V	40 to 80μm
х	30 μm
β*	90°

The symbols h, s, and  $\beta$  represent the height of the three-dimensional element, the height of the top portion of the three-dimensional element, and the angle shown in Fig. 4, respectively.

The obtained abrasive material was stamped out into a circular disk having a diameter of 110 mm to fabricate an abrasive disk. A circular rod of zirconia and an end surface of an optical connector ferrule were abraded with the use of this abrasive disk in the same manner as in Example 5. The change with time of the abraded amount of the circular rod of zirconia is shown in Fig. 16. The end surface of the optical connector ferrule was observed by an electron microscope, whereby a smooth surface was confirmed. A microscope photograph is shown in Fig. 18.

#### Example 7

An abrasive material was fabricated in the same manner as in Example 5 except that a mold sheet made of polypropylene and having recesses with a shape of inverted three-dimensional elements shown in Figs. 1 and 2 was used. In this abrasive material, the abrasive layer has a three-dimensional structure of a tetrahedral shape most closely packed as shown in Figs. 1 and 2. The dimensions are shown in Table 14.

Table 14

Symbol	Dimension (µm)	
h	63 μm	
S	50 μm	
О	190 μm	
р	190 μm	
α	90°	

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The obtained abrasive material was stamped out into a circular disk having a diameter of 110 mm to fabricate an abrasive disk. A circular rod of zirconia and an end surface of an optical connector ferrule were abraded with the use of this abrasive disk in the same manner as in Example 5. The change with time of the abraded amount of the circular rod of zirconia is shown in Fig. 16. The end surface of the optical connector ferrule was observed by an electron microscope, whereby a smooth surface was confirmed. A microscope photograph is shown in Fig. 19.

#### Example 8

An abrasive material was fabricated in the same manner as in Example 5 except that a mold sheet made of polypropylene and having recesses with a shape of inverted three-dimensional elements shown in Fig. 4 and being of a different type from the one used in Example 5 was used. In this abrasive material, the abrasive layer has a three-dimensional structure of a prismatic shape arranged in a stripe pattern as shown in Fig. 4. The dimensions are shown in Table 15.

Table 15

Symbol	Dimension (µm)
h	75 μm
S	50 µm
w	180 µm
р	180 µm
u	0 μm
β	100°

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The obtained abrasive material was stamped out into a circular disk having a diameter of 110 mm to fabricate an abrasive disk. A circular rod of zirconia and an end surface of an optical connector ferrule were abraded with the use of this abrasive disk in the same manner as in Example 5. The change with time of the abraded amount of the circular rod of zirconia is shown in Fig. 16. The end surface of the optical connector ferrule was observed by an electron microscope, whereby a smooth surface was confirmed. A microscope photograph is shown in Fig. 20.

# Comparative Example 7

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An abrasive material "Imperial Sign Diamond Lapping Film 3 Mil 9 Micron Type H" made by Minnesota Mining and Manufacturing Co., Ltd. was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk. A circular rod of zirconia and an end surface of an optical connector ferrule were abraded in the same manner as in Example 5 except that this abrasive disk was used. The change with time of the abraded amount of the circular rod of zirconia is shown in Fig. 16. The end surface of

the optical connector ferrule was observed by an electron microscope, whereby a rough surface was confirmed. A microscope photograph is shown in Fig. 21.

## Comparative Example 8

The abrasive slurry prepared in Example 5 was applied onto a polyester film having a thickness of 75  $\mu$ m by means of a knife coater and the solvent was removed by evaporation to form an abrasive layer having a thickness of 14  $\mu$ m. The abrasive layer was heated at 70°C for 24 hours and further heated at 100°C for 24 hours to harden the binder. The abrasive material obtained by cooling to room temperature was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

A circular rod of zirconia and an end surface of an optical connector ferrule were abraded in the same manner as in Example 6 except that this abrasive disk was used. The change with time of the abraded amount of the circular rod of zirconia is shown in Fig. 16. The end surface of the optical connector ferrule was observed by an electron microscope, whereby a rough surface was confirmed. A microscope photograph is shown in Fig. 22.

Example 9

An abrasive slurry was prepared by mixing the components shown in Table 16.

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Table 16

	Weight	Non-volatile	Weight after
Components	(g)	component (%)	drying (g)
Diamond abrasive grains	100.000	100.000	100.000
(particle size : 7 to 10 μm) "LS600F 7-10"			
(Lands Superabrasives, Co.)			
Resol-phenolic resin (non-volatile component	58.333	60.000	35.000
60%, water 20%, organic solvent 20%)			
Propylene glycol monomethyl ether	50.000	0.000	0.000
(PGM)			
Aerosol AY	1.000	50.000	0.500
(AMERICAN CYANAMID COMPANY)			
(as 50% MEK solution)			
Total	209.333	64.729	135.500

Abrasive grains/binder proportion = 2.86

Abrasive grains/(binder + additives) proportion = 2.82

A lamination binder was prepared by mixing the components shown in Table 17.

Table 17

		Non-volatile	Weight after
Components	Weight (g)_	component (%)	drying (g)
Epoxy resin "YD-128R"	96.000	100.000	96.000
(Toto Kasei Co., Ltd.)			
2-ethyl-4-methylimidazole	4.000	100.000	4.000
(2E4MZ)			
Total	100.000	100.000	100.000

The same mold sheet made of polypropylene as used in Example 1 was prepared. An abrasive slurry was applied onto the mold sheet by means of a roll coater and dried at 70°C for 5 minutes. The lamination binder was applied thereon and further a transparent polyester film having a thickness of 75 µm was superposed and pressed by a roll for lamination. Ultraviolet rays were radiated from the polyester film side to harden the lamination binder. Subsequently, the binder in the abrasive slurry was hardened by heating at 70°C for 24 hours.

The resultant was cooled to room temperature and the mold sheet was removed. The binder in the abrasive layer was hardened by further heating at 100°C for 24 hours. This abrasive material was stamped out into/a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

A circular rod of zirconia and an end surface of an optical fiber connector were abraded in the same manner as in Example 5 except that this abrasive disk was used. The change with time of the abraded amount of the circular rod of zirconia is shown in Fig. 16. The end surface of the optical fiber connector was observed by an electron microscope, whereby a smooth surface was confirmed. A microscope photograph is shown in Fig. 23.

# Example 10

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An abrasive material was fabricated in the same manner as in Example 9 except that the same mold sheet made of polypropylene as used in Example 6 was used. This abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk.

A circular rod of zirconia and an end surface of an optical fiber connector were abraded in the same manner as in Example 5 except that this abrasive disk was used. The change with time of the abraded amount of the circular rod of zirconia is shown in Fig. 16. The end surface of the optical fiber connector was observed by an electron microscope, whereby a smooth surface was confirmed. A microscope photograph is shown in Fig. 24.

#### Example 11

An abrasive material was fabricated in the same manner as in Example 9 except that a mold sheet made of polypropylene and having recesses with a shape of inverted three-dimensional elements shown in Fig. 3 was used. In this abrasive material, the abrasive layer has a three-dimensional structure of a pyramidal shape shown in Fig. 3 in which the top is truncated at a predetermined height. The dimensions are shown in Table 18.

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Table 18

Symbol	Dimension (µm)
h*	175 µm
s*	90 μm
0	250 μm
u	50 μm
y	150 µm
α*	30°

The symbols h, s, and  $\alpha$  represent the height of the three-dimensional element, the height of the top portion of the three-dimensional element, and the angle formed between two ridges of the three-dimensional element before the top is truncated, respectively.

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The obtained abrasive material was stamped out into a circular shape having a diameter of 110 mm to fabricate an abrasive disk. A circular rod of zirconia and an end surface of an optical fiber connector were abraded in the same manner as in Example 5 except that this abrasive disk was used. The change with time of the abraded amount of the circular rod of zirconia is shown in Fig. 16. The end surface of the optical fiber connector was observed by an electron microscope, whereby a smooth surface was confirmed. A microscope photograph is shown in Fig. 25.

From the graph shown in Fig. 16, it will be understood that the abrasive disks of Examples 5 to 11 exhibit a higher abrasive property and a longer product life than the abrasive disks of Comparative Examples 7 and 8. Also, by comparing Figs. 17 to 20 and 23 to 25 with Figs. 21 and 22, it will be understood that the abrasive disks of Examples 5 to 11 give a more smooth abraded surface than the abrasive disk of Comparative Example 7 which is a current product and the abrasive disk of Comparative Example 8 having an abrasive layer without a three-dimensional structure.

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As this invention may be embodied in several forms without departing from the spirit of essential characteristics thereof, the present embodiment is therefore illustrative and not restrictive, since the scope of the invention is defined by the appended claims rather than by the description preceding them, and all changes that fall within metes and bounds of the claims. or equivalence of such metes and bounds thereof are therefore intended to be embraced by the claims.

# What is Claimed is:

3.

 A method for producing an abrasive material having an abrasive layer of a three-dimensional structure, the method comprising the steps of:

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- (1) filling a mold sheet having a plurality of regularly arranged recesses, with an abrasive material coating solution containing abrasive grains, a binder, and a solvent, to a predetermined depth;
- (2) removing the solvent from the abrasive material coating solution in the recesses by evaporation;

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- (3) filling the recesses further with a binder;
- (4) laminating a base material on the mold sheet to bond the binder to the base material; and
  - (5) hardening the binder.

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2. The method according to claim 1, wherein said binder is hardened by ultraviolet radiation.

The method according to claim 1, wherein the binder contained in the

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abrasive material coating solution used in said step (1) is selected from the group consisting of phenolic resin, aminoplast resin, urethane resin, epoxy resin, acrylate resin, acrylated isocyanurate resin, urea-formaldehyde resin, isocyanurate resin, acrylated urethane resin, acrylated epoxy resin, resol-phenolic resin, polyester resin, vinyl resin, melamine resin and a mixture thereof.

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4. The method according to claim 1, wherein the binder used in said step (3) is selected from the group consisting of phenolic resin, aminoplast resin, urethane resin, epoxy resin, acrylate resin, acrylated isocyanurate resin, urea-formaldehyde resin, isocyanurate resin, acrylated urethane resin, acrylated epoxy resin, resol-phenolic resin, polyester resin, vinyl resin, melamine resin and a mixture thereof.

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5. An abrasive material for abrading an end surface of an optical fiber connector into a predetermined shape,

the abrasive material having a base material and an abrasive layer disposed on the base material,

the abrasive layer having a top layer comprising (1) an abrasive composite comprising abrasive grains dispersed within a binder and (2) a foot portion comprising a binder in the absence of abrasive particles,

the abrasive layer having a three-dimensional structure constructed with a plurality of regularly arranged three-dimensional elements having a predetermined shape.

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6. The abrasive material according to claim 5, wherein tops of said three-dimensional elements are constructed with points or lines parallel to a surface of the base material, and substantially all of said points or lines are located on a plane parallel to the surface of the base material.

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7. The abrasive material according to claim 5, wherein the concentration of the abrasive grains in the top layer of said abrasive layer exceeds a critical pigment volume concentration.

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8. The abrasive material according to claim 5, wherein the shape of said three-dimensional elements is a tetrahedral or pyramidal shape having ridges connected at a top.

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9. The abrasive material according to claim 5, wherein the three-dimensional elements have a height between about 2 micrometers and about 300 micrometers.

10. The abrasive material according to claim 9, wherein the three-dimensional elements height varies less than 20%.

11. The abrasive material according to claim 5, wherein the shape of said three-dimensional elements is a prismatic shape having ridges parallel to a surface of the base material at a top.

5 12. The abrasive material according to claim 5, wherein the abrasive grains size is between about 0.01 and about 1 micrometer.

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13. The abrasive material according to claim 5, wherein the abrasive grains size is between about 0.5 and about 20 micrometer.

14. The abrasive material according to claim 5, wherein the abrasive grains nominal size is between about 2 and about 4 micrometers.

- The abrasive material according to claim 5, wherein the abrasive grains nominal size is between about 7 and about 10 micrometers.
  - 16. The abrasive material according to claim 5, wherein the abrasive grains maximum size is about 16 micrometers.
  - 17. The abrasive material according to claim 5, wherein the abrasive grains average size is between about 7.5 and about 9.5 micrometers.
    - 18. The abrasive material according to claim 5, wherein said binder is selected from the group consisting of phenolic resin, aminoplast resin, urethane resin, epoxy resin, acrylate resin, acrylated isocyanurate resin, urea-formaldehyde resin, isocyanurate resin, acrylated urethane resin, acrylated epoxy resin, resol-phenolic resin, polyester resin, vinyl resin, melamine resin and mixtures thereof.
- The abrasive material according to claim 5, wherein said abrasive grains are selected from the group consisting of fused aluminum oxide, heat-treated aluminum oxide, silicon carbide, alumina zirconia, garnet, diamond, cubic boron nitride, silica,

cerium oxide, sol-gel aluminum oxide, chromium oxide, zirconia, iron oxide and mixtures thereof.

- The abrasive material according to claim 5, wherein said base material is flexible to be particularly suited for spherically abrading an end surface of an optical fiber connector.
- 21. The abrasive material according to claim 20, which is capable of providing an optical fiber connector end surface having a connection loss of not more than 1.0dB.

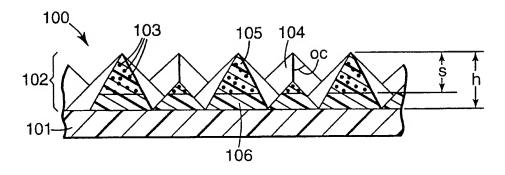


Fig. 1

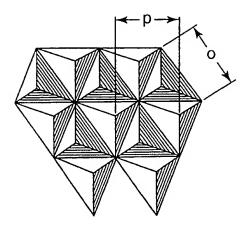


Fig. 2

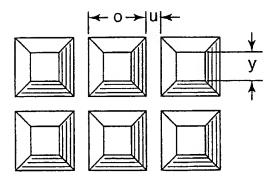


Fig. 3

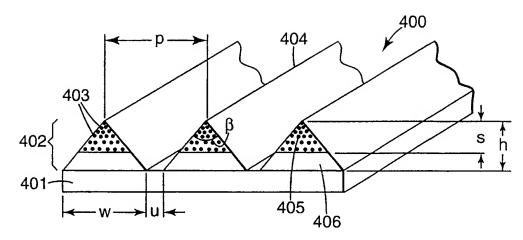


Fig. 4

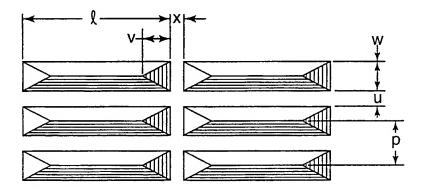
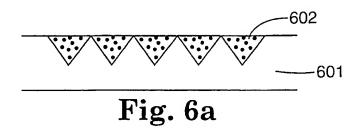


Fig. 5



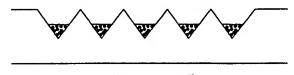


Fig. 6b

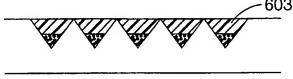


Fig. 6c

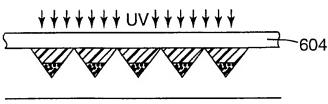
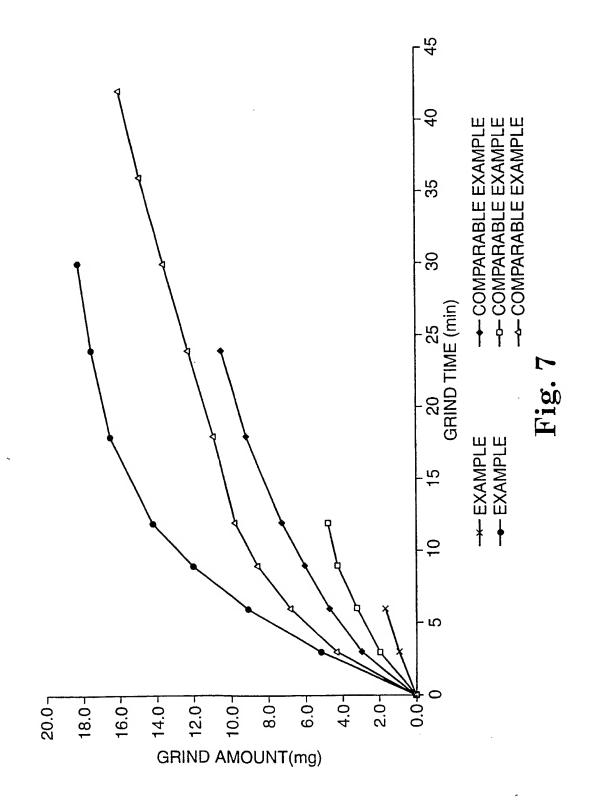
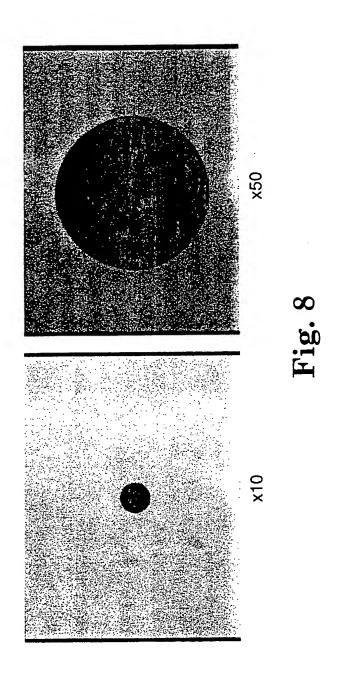


Fig. 6d

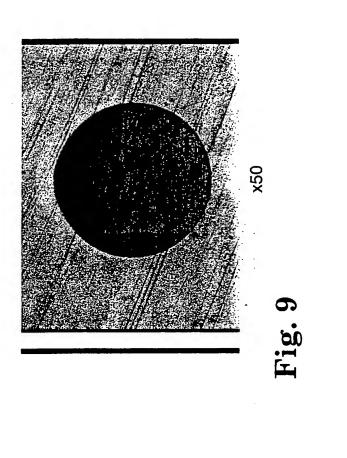


Fig. 6e



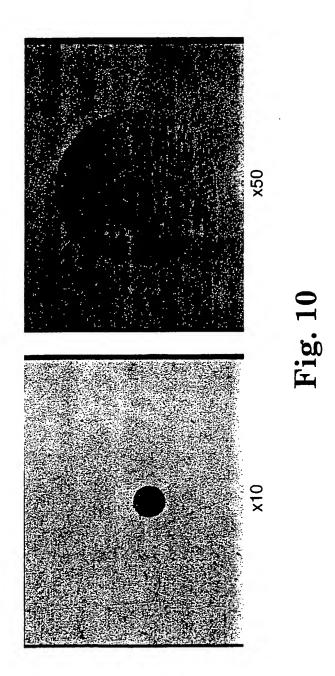


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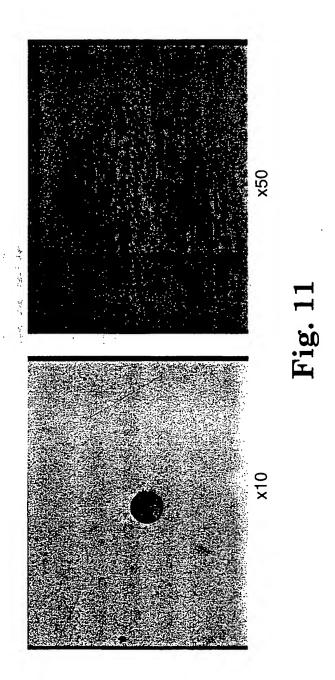


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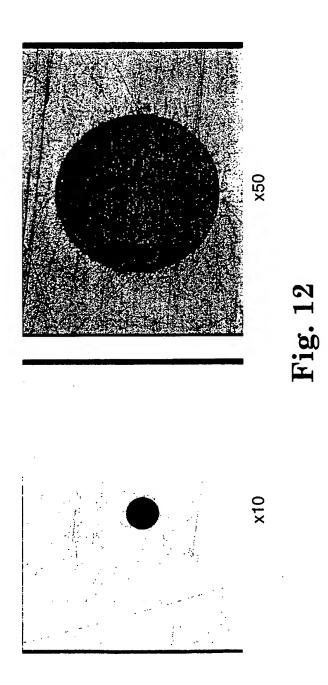
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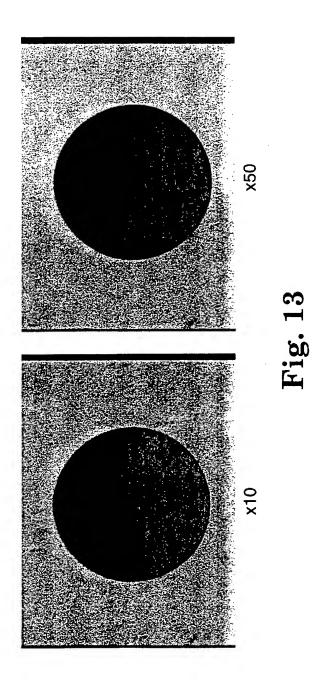
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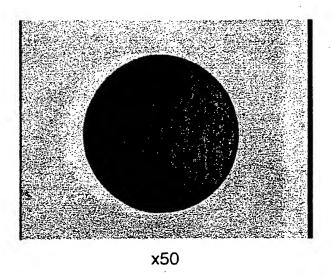


Fig. 14

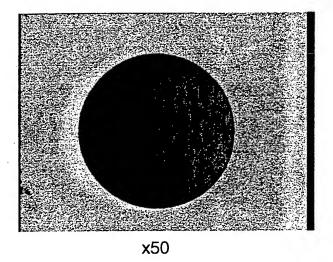
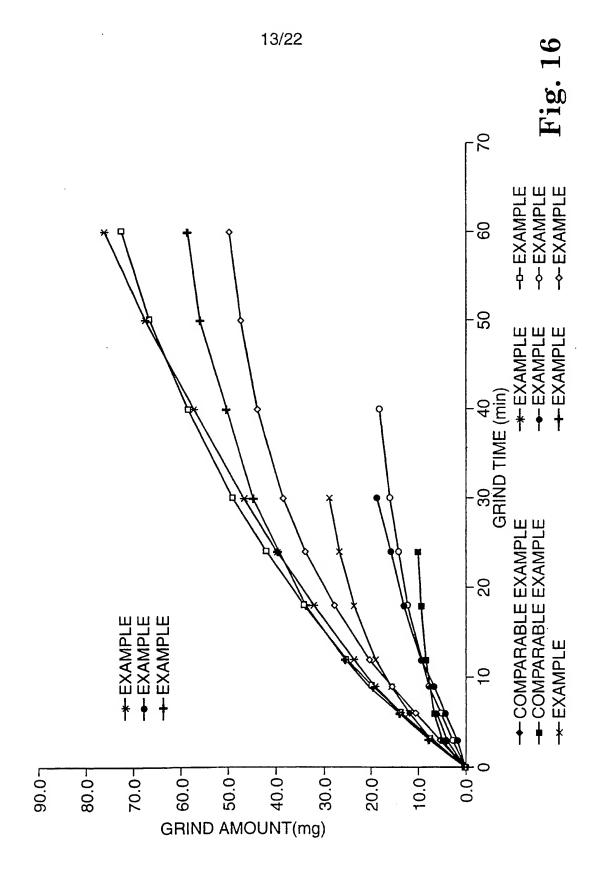
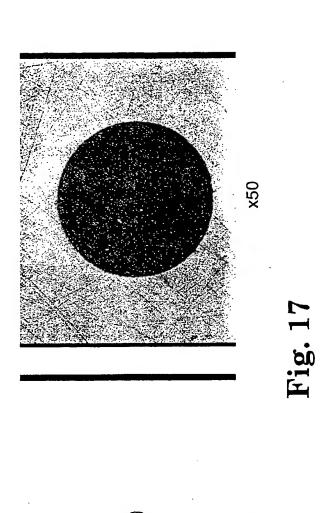


Fig. 15

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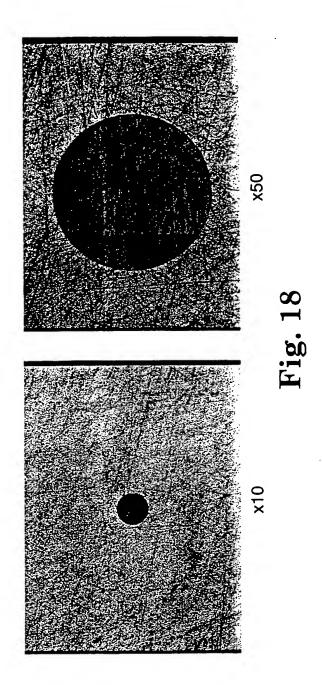


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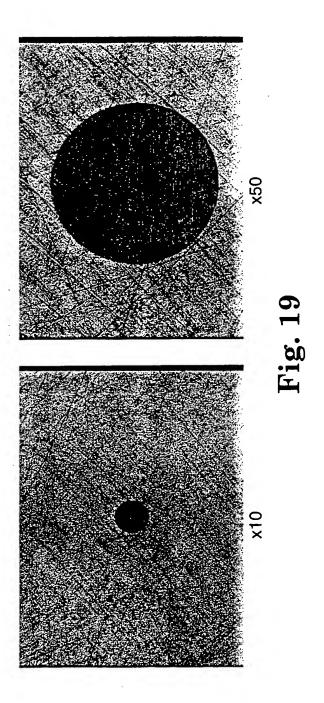


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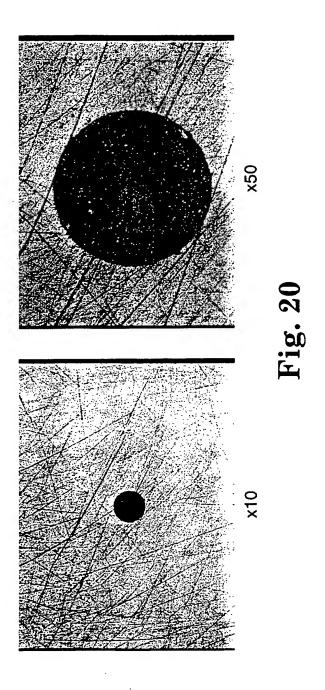
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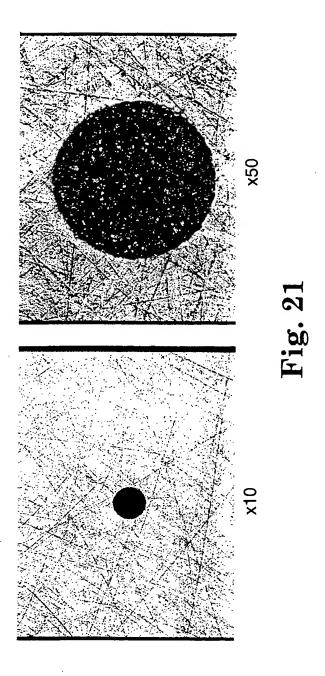
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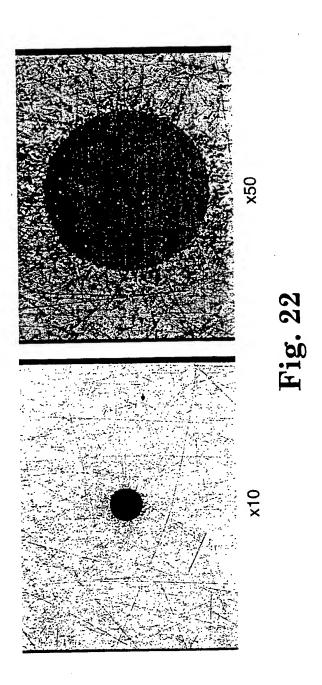
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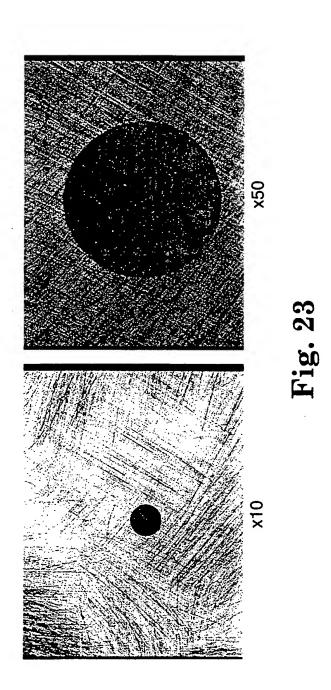
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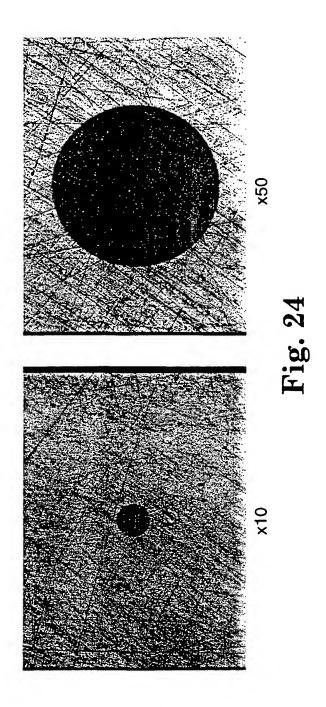
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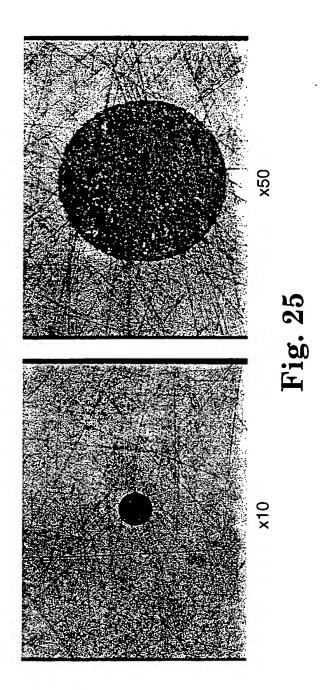
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### INTERNATIONAL SEARCH REPORT

Intern :al Application No PCT/US 00/35355

A. CLASSII IPC 7	FICATION OF SUBJECT MATTER B24D11/00 B24D3/28								
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EPO-In	ternal								
C. DOCUMENTS CONSIDERED TO BE RELEVANT									
Category *	Citation of document, with indication, where appropriate, of the	e relevant passages	Relevant to claim No.						
X	EP 0 664 187 A (DAINIPPON PRINT 26 July 1995 (1995-07-26) page 3, line 5 page 4, line 26-33 claims 5,17	1-21							
X	WO 99 59778 A (3M INNOVATIVE PR CO) 25 November 1999 (1999-11-2 page 21, line 30 -page 22, line	5-21							
A	US 5 152 917 A (MINNESOTA MININMANUFACTURING CO)) 6 October 1992 (1992-10-06) the whole document & WO 92 13680 A 20 August 1992 (1992-08-20) cited in the application	NG AND	1-21						
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### INTERNATIONAL SEARCH REPORT

Intern: :: al Application No PCT/US 00/35355

		PC1703 00/35355		
C.(Continua	ation) DOCUMENTS CONSIDERED TO BE RELEVANT			
Category °	Citation of document, with indication, where appropriate, of the relevant passages		Relevant to claim No.	
Ρ,Χ	WO 00 13853 A (3M INNOVATIVE PROPERTIES CO) 16 March 2000 (2000-03-16) page 23, line 28 -page 24, line 1; figures 2,4		5-21	
	·			

2

# INTERNATIONAL SEARCH REPORT

Information on patent family members

Intern 181 Application No PCT/US 00/35355

Patent document cited in search repor	t	Publication date		Patent family member(s)	Publication date
EP 0664187	A	26-07-1995	WO	9427787 A	08-12-1994
LI 0004107	••	20 07 2000	DE	69326774 D	18-11-1999
			DE	69326774 T	21-06-2000
			JP	2980682 B	22-11-1999
			KR	165625 B	01-02-1999
			US	5709598 A	20-01-1998
			ÜS	5868806 A	09-02-1999
			US	5908476 A	01-06-1999
WO 9959778		25-11-1999	AU	3779799 A	06-12-1999
			EP	1079954 A	07-03-2001
US 5152917	 А	06-10-1992	AT	137154 T	15-05-1996
			AU	661473 B	27-07-1995
			AU	1240392 A	07-09-1992
			BR	9205596 A	26-04-1994
		•	CA	2100059 A	07-08-1992
			CN	1269277 A	11-10-2000
			CN	1064830 A	30-09-1992
			CZ	9301581 A	16-02-1994
			DE	69210221 D	30-05-1996
			DE	69210221 T	09-01-1997
			EP	0570457 A	24-11-1993
			ES	2086731 T	01-07-1996
			HK	1006688 A	12-03-1999
			HU	68648 A	28-07-1995
			JP	6505200 T	16-06-1994
			KR	216381 B	16-08-1999
			MX	9200306 A	01-09-1992
			RU	2106238 C	10-03-1998
			SG	73390 A	20-06-2000
			US	5304223 A	19-04-1994
			WO	9213680 A	20-08-1992
			US	5378251 A	03-01-1995 
WO 0013853	Α	16-03-2000	AU	5546199 A	27-03-2000
			TW	411306 B	11-11-2000

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